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# EFFECT OF VACUUM ON THE CREEP-RUPTURE PROPERTIES OF SINTERED ALUMINUM AND COMMERCIAL ALUMINUM

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## ABSTRACT

In an investigation of the effect of environment on creep-rupture properties, it has been found that vacuum increases the life of sintered aluminum and improves the ductility of commercial purity aluminum at 400°, 500°, and 600°C. Metallographic studies show that the environmental effects can be related to the appearance of porosity in the dispersion-hardened material and grain boundary fissures in the unmodified aluminum. It is concluded that the improvements conferred by the vacuum environment are a result of its control of the hydrogen content of both materials.


## PROBLEM STATUS

This report completes one phase of the problem; work on other aspects of the problem is continuing.

## AUTHORIZATION


NRL Problem M01-09
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# EFFECT OF VACUUM ON THE CREEP-RUPTURE PROPERTIES OF SINTERED ALUMINUM AND COMMERCIAL ALUMINUM

## INTRODUCTION

Previous investigations have shown that the high-temperature creep-rupture properties of nickel and nickel-base alloys in air may be either higher or lower than in vacuum (1,2). A mechanism to explain this reversal of the effect of environment was proposed. The mechanism is based upon two competing processes: oxidation, which hardens and strengthens a metal, prolongs its rupture life, while surface adsorption of gas, by lowering the energy necessary to propagate a crack, accelerates rupture. The process which controls is determined by temperature and strain rate.

In the present investigation aluminum was selected for further study in the expectation that there would be no strengthening by oxidation, since aluminum is relatively immune to this type of attack, and that the study of the influence of gas adsorption on crack propagation would be simplified. The two materials used for the investigation were sintered aluminum, containing a dispersion of its oxide and, for comparison, commercially pure aluminum. Creep-rupture tests were conducted in air and in vacuum, and the environmental effects observed are discussed in terms of the mechanism of fracture.

## EXPERIMENTAL PROCEDURE

The commercially pure aluminum, designated 1100 alloy, contains a nominal 99 percent Al. It is the base for the sintered alloy, designated M-257, which contains 7.8 percent oxide. The material was furnished as extruded bar of 1 inch diameter. Conventional rod-type specimens of 1/2 inch diameter were used; the gage lengths for the 1100-alloy rods were 1 inch and for the sintered aluminum specimens the lengths were 2 inches.

Conventional creep-rupture equipment was used which incorporated vacuum chambers evacuated to $1\times10^{-5}$ torr (3). Creep rates were measured from movements of the lever arm.

Some of the tests were conducted on specimens in the as-received condition, while others were first annealed for 2 hours at 600°C in a vacuum of $1\times10^{-5}$ torr.

## RESULTS

### Hydrogen Analyses

During the creep-rupture testing on both materials, evidence was found for a so-called "vacuum-anneal" effect. Exposure to vacuum, either before or during the test, affected the properties of both test materials in a way which was indicative of a reduction in their gas contents. To determine the cause of the vacuum-anneal effect, hydrogen analyses were made on both materials by vacuum fusion. As shown by the results in Table 1, it is clear, despite some scatter, that the original (as-received) hydrogen content of the M-257, which was approximately 5 ppm, is partially reduced by an air anneal and more completely reduced in vacuum. The completeness of removal is improved by higher temperatures and longer times to an ultimate value of about 1 ppm residual hydrogen.

Table 1
Hydrogen Contents of M-257 Alloy

| Thermal History | | Test Environment | Contents (ppm) |
|---|---|---|---|
| Temp. (°C) | Time (hr) | | |
| As received | | - | 4.7 |
| As received | | - | 5.6 |
| As received | | - | 6.1 |
| 600 | 2 | Vac | 1.1 |
| 600 | 2 | Vac | 1.6 |
| 600 | 46 | Vac | 1.0 |
| 600 | 139 | Vac | 0.6 |
| 600 | 1054 | Vac | 1.1 |
| 400 | 105 | Vac | 3.0 |
| 600 | 3.6 | Air | 3.6 |
| 600 | 2 | Air | 3.1 |
| 600 | 11.5 | Air | 5.6 |
| 600 | 67 | Air | 2.2 |

For the 1100 alloy, it was not possible to correlate hydrogen concentration with thermal history because the blank-off for the specimen size used was 0.5 ppm, which is comparable with the content of the material. However, the sensitivity of the rupture and of the ductility to vacuum exposure indicates that gas is extracted from this material also.

Creep-Rupture of the 1100 Alloy

In Fig. 1 it is seen that at 400 C there is a reversal in the environmental effect for those specimens of the 1100 alloy which have not been vacuum annealed. In short time tests, the longer lifetime in air than in vacuum is not an example of oxidation hardening. To account for the shorter lifetime in vacuum than in air, it is to be noted that the specimens in these short time tests are characterized by tensile-type failures, Fig. 2(a). The extraction of gas, which blocks plastic flow, would promote faster necking down and failure of the specimens in vacuum. At longer times, where ruptures in air are of the intergranular type with low reduction of area, Fig. 2(b), degassing in vacuum suppresses grain boundary failure and prolongs the lifetime, Fig. 2(c). If the gas is more completely removed by a prior vacuum anneal at 600 C, there is 100-percent reduction of area even in an air test, Fig. 2(d).

It is shown by the lower curve of Fig. 1 that specimens which have been vacuum annealed are not appreciably affected by the test environment. The vacuum at 400°C evidently does not remove additional gas after the vacuum anneal at 600°C. The creep-rupture curve for the vacuum-annealed specimens is substantially lower than for those specimens which have not been vacuum annealed. Evidently, the 600°C anneal coarsened the grain size of the specimens and reduced the creep resistance.

Creep curves such as Fig. 3 demonstrate that vacuum prolongs rupture life by postponing the initiation of third-stage creep. The elimination of gas evidently suppresses the formation of grain boundary cracks, which cause an acceleration of the creep rate.

At 500 C, Fig. 4, the specimens which have not been heat treated are appreciably stronger in vacuum than in air since at this increased temperature the gas is more completely removed by the vacuum environment of the test. At this temperature the

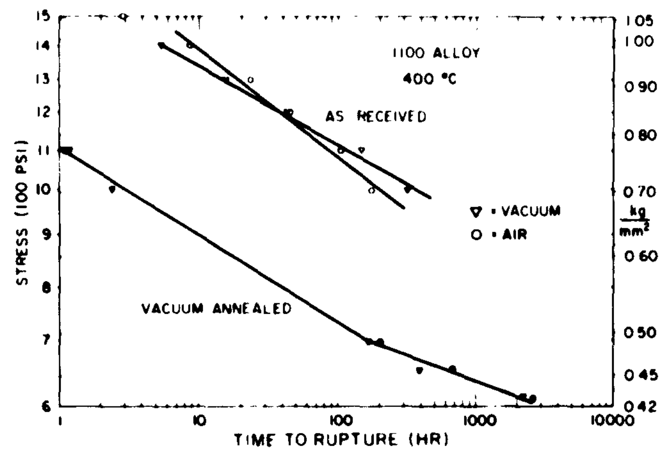


Fig. 1 - Creep-rupture of the as-received and of the vacuum-annealed 1100 alloy at 400°C in air and in vacuum

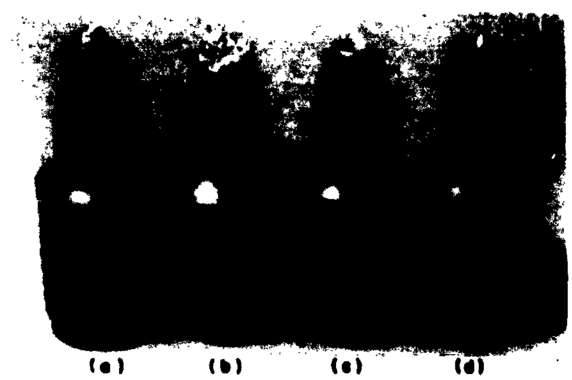


Fig. 2 - Specimens of the 1100 alloy ruptured at 400°C: (a) tested in air in the as-received condition at 1400 psi, rupture life - 7 hr; (b) tested in air in the as-received condition at 1000 psi, rupture life - 177 hr; (c) tested in vacuum in the as-received condition at 1000 psi, rupture life - 318 hr; and (d) vacuum annealed prior to testing in air at 650 psi, rupture life - 667 hr

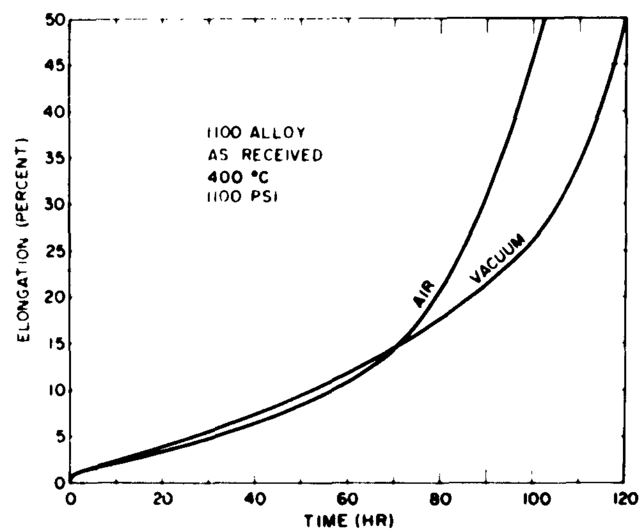


Fig. 3 - Creep curves for the as-received 1100 alloy at 400°C and 1100 psi in air and in vacuum

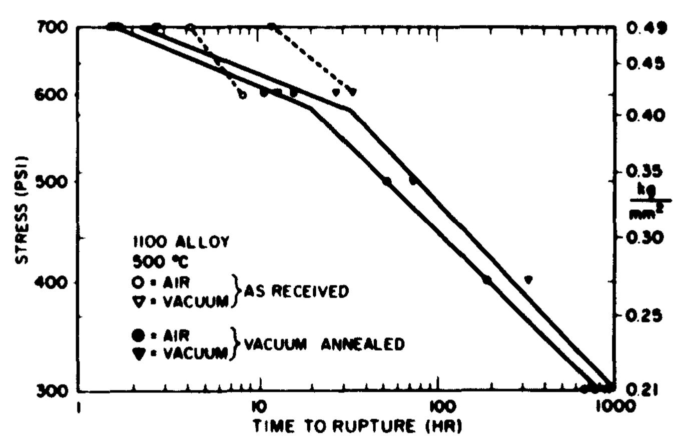


Fig. 4 - Creep-rupture of the as-received and vacuum-annealed 1100 alloy at 500°C in air and in vacuum

vacuum-annealed specimens also showed an atmosphere effect. Thus, even after a vacuum anneal at 600°C, exposure to the vacuum environment at 500°C evidently results in continued extraction of gas.

At 600°C it was not possible to compare rupture lives in the two environments because the specimens in vacuum were elongated to the limit of the equipment without rupturing. In Fig. 5, where the time to reach an elongation of 37.5 percent is plotted versus stress, it is seen that the creep rate was appreciably lower in vacuum than in air. In Fig. 6 the vacuum effect is clearly shown by the specimens. With no pretreatment those tested in air ruptured with a flat, creep-type fracture, Fig. 6(b), but were more ductile in vacuum, Fig. 6(d). Even when tested in air, they are ductile if they have been vacuum annealed before the test, Fig. 6(c). As demonstrated by the micrographs of Fig. 7, exposure to vacuum improves the grain boundary cohesion. The fissures, or grain boundary separations, in Fig. 7(a) are virtually eliminated by a vacuum anneal, Fig. 7(b).

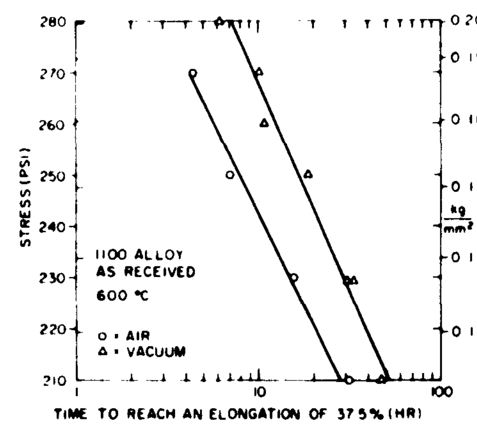


Fig. 5 - Time to reach 37.5-percent elongation for the 1100 alloy at 600°C in air and in vacuum

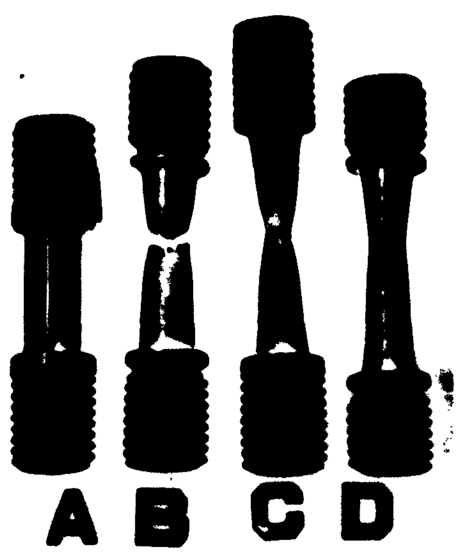


Fig. 6 - Specimens of the 1100 alloy tested at 600°C and 230 psi: (a) as machined; (b) as received and tested in air; (c) vacuum annealed and tested in air; and (d) as received and tested in vacuum

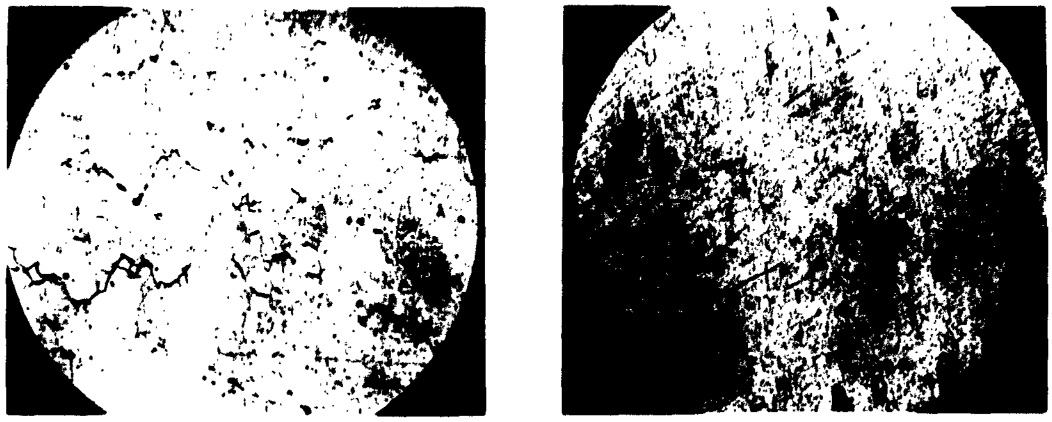

Fig. 7 - Micrographs of the 1100 alloy tested in air at 250 psi and 600 C (a) as received, rupture life = 6.8 hr; and (b) vacuum annealed before test, rupture life = 20.8 hr

## Creep-Rupture of the M-257 Alloy

In Fig. 8 the effect of vacuum on the sintered material is shown in three ways. First, specimens in the as-received condition last longer in vacuum than in air, especially in low-stress, long-time tests. Second, when tested in air, the rupture life is greater if testing is preceded by a vacuum anneal. Third, after a vacuum anneal, the rupture life is greater in vacuum than in air. The second effect is clearly a result of the extraction of hydrogen by vacuum. The interpretation of the third effect is not as straightforward. It could signify either a continued extraction, in the vacuum at 400°C, of the residual hydrogen remaining from the 600°C vacuum anneal or a reaction of the metal with air which could facilitate crack propagation.

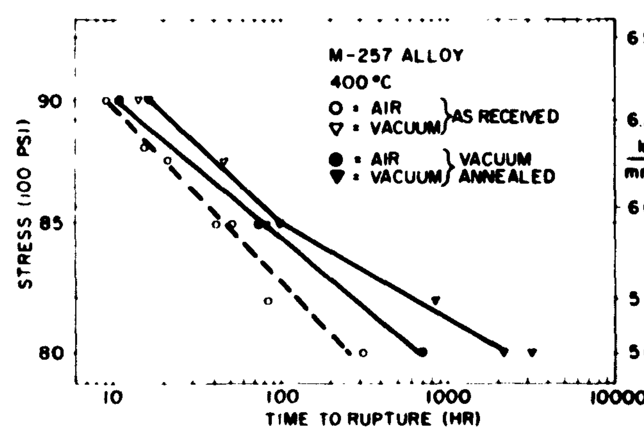


Fig. 8 - Creep-rupture of the as-received and of the vacuum-annealed M-257 allpy at 400°C in air and in vacuum

A consideration of the specimens tested at 8000 psi, Fig. 8, sheds some light on this question. A comparison of the microstructures of the two as-received specimens shows that the porosity of the air-tested specimen, Fig. 9(a), is slightly greater than the one in vacuum, Fig. 9(b). If, however, testing is preceded by a vacuum anneal at 600°C, the porosity is greatly reduced, Fig. 9(c). Since this porosity is entirely absent in the M-257 alloy in the as-received condition, Fig. 9(d), one concludes that the porosity is introduced during creep and that the amount of it depends on the hydrogen content. But the specimen in Fig. 9(c), which displays little porosity, and, therefore, has a low hydrogen content, failed sooner when tested in air than the one in Fig. 9(b) which was tested in vacuum. These observations indicate that reactions with atmospheric gases reduced the life of the specimen in Fig. 9(c), which had a low hydrogen content, by furnishing a gaseous component which could adsorb on crack surfaces and lower the energy necessary for crack propagation.

Additional evidence for the effect of hydrogen content is furnished by an examination of rupture surfaces. The fracture in air after 41 hours, Fig. 10(a), has a very brittle appearance. When failure took place after the longer time of 84 hours, the rupture has a smoother, less brittle appearance, Fig. 10(b). If the specimen is vacuum annealed, however, the fracture surfaces are quite smooth, as shown in Fig. 10(c). Heating the material in air at 400 C removes hydrogen, but it is extracted faster in vacuum at 600°C.

At 500 C, Fig. 11, prolongation of rupture life in vacuum is again demonstrated, especially in longer time tests. Again, the appearance of the fracture surfaces in Fig. 12 indicates that gas extraction is faster in vacuum than in air. Typical creep curves are shown in Fig. 13. One cannot use data from curves such as these to determine the effect of environment on the elongation at fracture. Since the elongations are very low, never much greater than one percent, the differences between air and vacuum specimens would be well within the uncertainty of the measurement. Nor is it possible to measure elongation at rupture on the specimens because the broken halves cannot be fitted together.

At 600°C, although the scatter is much more pronounced, the effects, especially at long times, are large enough so that there is no doubt that they exist. The rupture lives in Fig. 14 are longer in vacuum than in air. Vacuum annealing, before testing in air, again improves the life, but not as much as the improvement obtained by testing in vacuum. Additional evidence for the effect of gas extraction is found in a study of the microstructure. In Fig. 15(a), the porosity resulting from heating in air at 600°C for 16 hours is less than that of the 400°C specimen in Fig. 9(a). After 1054 hours in vacuum at 600°C, Fig. 9(b), the porosity is virtually eliminated.

A search was made for cracks, by metallographic and crack detection methods, in specimens which were both ruptured and interrupted prior to failure. After failure in air or in vacuum, no cracks were found. Evidently, once a crack is initiated, it is propagated rapidly to failure. This observation would explain why creep curves of this material display virtually no third stage of creep.

## DISCUSSION

It appears quite clear that heating in vacuum removes hydrogen and improves the properties of both the commercial purity aluminum and the oxide-modified alloy. This conclusion is derived from the fact that a prior vacuum anneal improves the ductility of the unmodified aluminum and the creep-rupture strength of the dispersion-hardened material when the testing on both alloys is carried out in air. Although correlations of hydrogen content with heat treatment and mechanical properties were found only for the sintered alloy – the changes in hydrogen content for the unmodified aluminum were too small for analysis – the response of the unmodified material to vacuum indicates that its properties are also being affected by the removal of a gas, most likely hydrogen. Similar conclusions

(a)

(b)

Fig. 9 - Micrographs of the M-257 alloy tested at 400°C and 8000 psi: (a) tested in air in the as-received condition, rupture life – 200 hr; (b) tested in vacuum in the as-received condition, interrupted after 3000 hr; (c) vacuum annealed prior to testing in air, rupture life – 700 hr; and (d) as received

(c)

(d)

Fig. 9 (Continued) - Micrographs of the M-257 alloy tested at 400°C and 8000 psi: (a) tested in air in the as-received condition, rupture life - 200 hr; (b) tested in vacuum in the as-received condition, interrupted after 3000 hr; (c) vacuum annealed prior to testing in air, rupture life - 700 hr; and (d) as received

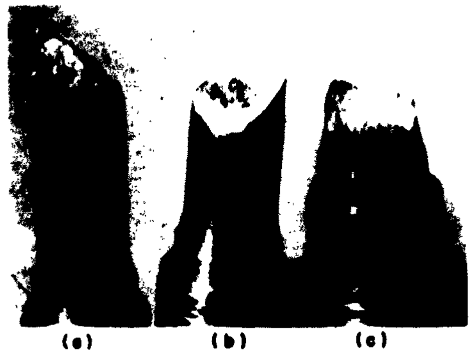


Fig. 10 - Specimens of the M-257 alloy ruptured at 400°C: (a) tested in air in the as-received condition at 8500 psi, rupture life - 41 hr; (b) tested in air in the as-received condition at 8200 psi, rupture life - 84 hr; and (c) vacuum annealed prior to testing in vacuum at 8500 psi, rupture life - 100 hr

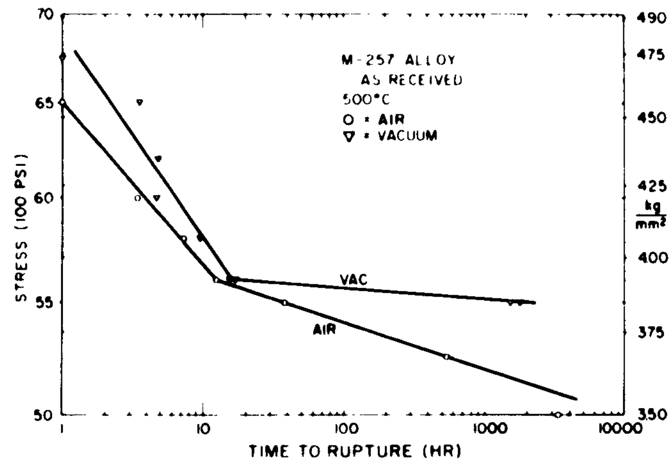


Fig. 11 - Creep-rupture of the as-received M-257 alloy at 500°C in air and in vacuum

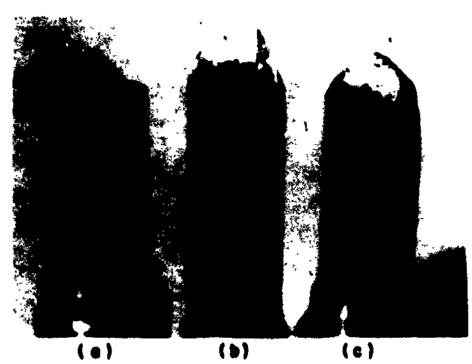


Fig. 12 - Specimens of the M-257 alloy ruptured at 500°C in the as-received condition: (a) tested in air at 6000 psi, rupture life - 3 hr; (b) tested in air at 5600 psi, rupture life - 12 hr; and (c) tested in vacuum at 5600 psi, rupture life - 15 hr

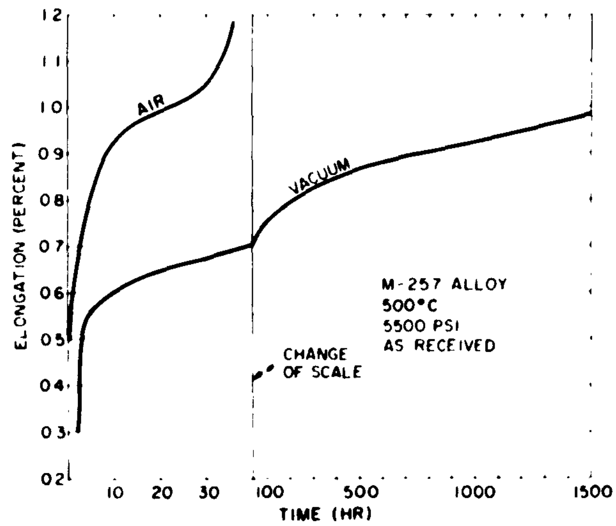


Fig. 13 - Creep curves for the as-received M-257 alloy at 500°C and 5500 psi

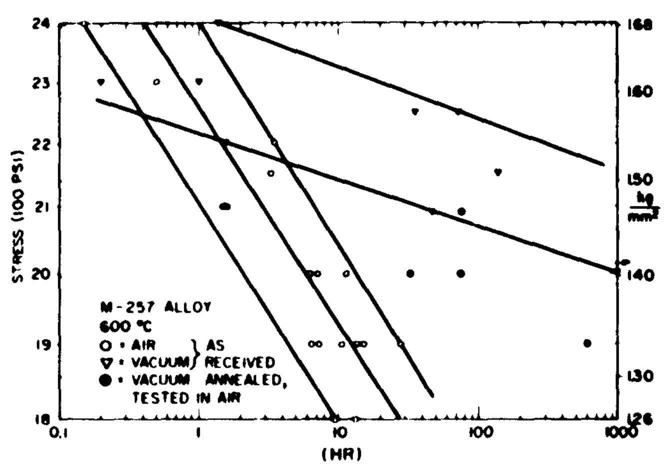


Fig. 14 - Creep rupture of the as-received and of the vacuum-annealed M-257 alloy at 600°C

(a)

(b)

Fig. 15 - Micrographs of the M-257 alloy tested at 600°C in the as-received condition: (a) in air at 1900 psi, rupture life – 16 hr; and (b) in vacuum at 2000 psi, interrupted at 1054 hr

were reached by O'Dette (4) for aluminum, and by Kessler (5) and Cremens, Bryan, and Grant (6) for SAP. In the previously mentioned investigations blister formation was attributed to hydrogen.

It is not the purpose of this paper to devote space to a discussion of the mechanism whereby hydrogen facilitates the cracking of the alloys used in this study. The literature is replete with treatments of this subject for a great variety of metals.

It is not clear whether, superimposed on this annealing effect, there is an atmospheric effect arising from the reaction of these materials with air. There is no evidence for such a reaction for the unmodified aluminum, but there is for the dispersion-strengthened material. That is, the specimen whose porosity was reduced by a vacuum anneal failed in air before the specimen tested in vacuum. Reaction with atmospheric moisture might introduce enough hydrogen to accelerate the formation of a crack. In support of this reaction, the work of Blackburn and Gulbransen can be cited (7). They reported an increase of the hydrogen content of aluminum when it is heated at 600°C in water vapor.

According to the model usually used to explain hydrogen effects, only enough gas to cover the crack surfaces is necessary. In our specimen only one crack is initiated. The small amount of hydrogen necessary to cover the two surfaces would not be detectable by vacuum fusion analysis.

## CONCLUSIONS

From a study of the creep-rupture properties of commercial purity aluminum and of aluminum with a dispersion of its oxide at 400°, 500°, and 600°C in air and in vacuum, the following conclusions have been reached:

1. The hydrogen content of the sintered material is reduced faster by an anneal in vacuum than in air. As a result of this degassification, specimens annealed in or tested in vacuum have longer rupture lives than those which have not been exposed to vacuum.

2. Metallographic investigation has shown that porosity is developed during creep of sintered material containing hydrogen. Diminishment of the porosity may be correlated with thermal treatments which reduce the hydrogen content.

3. There is indirect, but still inconclusive, evidence that the rupture life of the oxide-modified material is reduced by a reaction with the water vapor in air.

4. Although vacuum annealing does not appear to reduce measurably the hydrogen content of the unmodified alloy, observations of improvement in ductility by suppression of grain boundary cracking indicate that a contaminant, probably hydrogen, is extracted during the anneal.

## ACKNOWLEDGMENTS

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